PAUUU1 28/5/91
Section 29

AUSTRALIA

Patents Act 1990

PATENT REQUEST: STANDARD PATENT/PATENT OF ADDITION

We, being the persons identified below as the Applicant, request the grant of a patent to the person identified below as the Nominated Person, for an invention described in the accompanying standard complete specification.

Full application details follow.

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[54] Invention Title: PEARLESCENT CONCENTRATE

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ASSOCIATED PROVISIONAL APPLICATION(S) DETAILS

[60] Application Number(s) and Date(s)

PM5046 FILED 13 APRIL 1994

Drawing number recommended to accompany the abstract .....

By our Patent Attorneys,
WATERMARK PATENT & TRADEMARK ATTORNEYS

.....

DATED this 12th day of April 1995.

Darryl B. Mischlewski

Registered Patent Attorney

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PEARLESCENT CONCENTRATE

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(57)

A low viscosity pearlescent concentrate comprising

(i) 60 - 80% wt of a lauryl sulphate salt;

(ii) 12 - 20% wt of a fatty acid amide containing a quaternary ammonium group and a carboxyl radical; and

(iii) 6 - 15% wt of a mixture of ethylene glycol monostearate and ethylene glycol distearate, preferably in the ratio of 60 to 40% by weight distearate to 40 to 60 % by weight monostearate;

for use in cosmetic preparations, hair and washing or shower preparations. The concentrates have a range of pearlescent sheens, are not contaminated by nitrosamine or 1,4-dioxane and are preferably prepared without the addition of a viscosity modifier. The concentrates are also preferably translucent in the finished product enabling the colour of the finished product to be readily seen.

Preferably the concentrates are prepared by

(a) dissolving the mixture of ethylene glycol monostearate and ethylene glycol distearate in a hot solution containing at least 40% of the total amount of lauryl sulphate salt; and

(b) precipitating crystals in the solution by rapidly cooling the solution to a temperature 15 to 30° above ambient temperature followed by slowly cooling the solution to ambient temperatures.

AUSTRALIA

Patents Act 1990

ORIGINAL
COMPLETE SPECIFICATION
STANDARD PATENT

Application Number:

Lodged:

Invention Title: PEARLESCENT CONCENTRATE

The following statement is a full description of this invention, including the best method of performing it known to us :-

PEARLESCENT CONCENTRATE

This invention relates to pearlescent concentrates for use in cosmetic preparations, hair and washing or shower preparations.

Pearlescent preparations are known. Generally they are surfactant compositions containing substances which precipitate on cooling to form fine, nacreous crystals which remain dispersed in the preparation to provide a pearly, aesthetically attractive appearance. Known pearlescents are mono-esters or di-esters of ethylene glycol, propylene glycol and oligomeric alkylene glycols, fatty acids and mono-alkanolamides of fatty acids and lower alkanolamines.

Pearlescents vary in the quality of the sheen produced, ranging from a pearl-like lustre to a more metallic sheen. Known pearlescents also give a matt or opaque effect in the product to which they are added, with the result that the colour of the finished product is washed out.

Further, prior art pearlescent compositions have commonly contained mono- or di-alkanolamides to improve the pearlescent properties of the composition. (See for example GB 1230413, US477038 and US4824594). However, as alkanolamines and derivatives thereof have been suspected of participating in the formation of nitrosamines, the use of alkanolamides is no longer desirable.

In addition, pearlescent concentrates, particularly those free of alkanolamides, are commonly highly viscous and their low flowability and pumpability makes them difficult to handle when preparing cosmetic or other preparations to which they are added.

The use of viscosity modifiers in pearlescent concentrates has been proposed to increase the flowability and pumpability of concentrates. In Australian Patent No. 625976 and Australian Patent Application No. 11744/92, the use of low molecular weight, polyhydric alcohols is proposed to reduce the viscosity of the pearlescent concentrate to improve its flowability and pumpability.

One low viscosity product, without viscosity modifiers, is known, comprised of a blend of sodium lauryl ether sulphate and coco amido lauryl

ether sulfate. However the white colour of this pearlescent reduces the colour of the products to which it is added.

It is an object of this invention to provide low viscosity pearlescent concentrates with a range of pearlescent sheens, without contamination by nitrosamine or 1,4 dioxane and which preferably do not require the addition of a viscosity modifier and preferably which is translucent in the finished product, enabling the colour of the finished product to be readily seen.

To this end the present invention provides a pearlescent concentrate comprising

(i) 60 - 80% wt of a lauryl sulphate salt;

(ii) 12 - 20% wt of a fatty acid amide containing a quaternary ammonium group and a carboxyl radical; and

(iii) 6 - 15% wt of a mixture of ethylene glycol monostearate and ethylene glycol distearate.

The concentrates are prepared under conditions which result in a flowable product with a pearl-like sheen, without the need for a viscosity modifier.

The present invention is partially predicated on the discovery that the ratio of mono- to di-esters in the ethylene glycol stearate affects the viscosity of the pearlescent. Ethylene glycol stearates with a ratio of 40% to 60% mono-ester to 60% to 40% di-ester yield low viscosity pearlescents without addition of a viscosity modifier. Preferably the mixture of ethylene glycol monostearate and ethylene glycol distearate comprises 40% to 60% ethylene glycol distearate and 60% to 40% ethylene glycol stearate by weight and more preferably the pearlescent concentrate of the present invention contains 44 to 54% by weight ethylene glycol monostearate. Particularly suitable ethylene glycol stearates are Croda EGMS (experimental) and EGMS 3127. Croda EGMS (experimental) is a ethylene glycol monostearate containing 40% distearate and 60% monostearate, available from Croda Singapore Pte Ltd. EGMS 3127 is a non-self-emulsifying glycol monostearate containing 56% distearate and 44% monostearate, also available from Croda Singapore Pte Ltd.

The pearlescent concentrate of the present invention preferably has a viscosity of less than 10000 cps, and hence high flowability and good pumpability. More preferably the concentrate has a viscosity of between 1750 and 6000 cps.

In another aspect of this invention, it has been discovered that the particle size distribution of the nacreous crystals influences pearlescence. To this end a preferred embodiment of the present invention provides a pearlescent concentrate composed of fine crystals dispersed in a liquid phase, said crystals preferably having a mean particle size of in the range of 7 to 13 microns and a particle size distribution such that at least 85% of the particles are sized between 5 to 20 microns. More preferably at least 90% of the particles are sized between 5 to 20 microns.

In a still further aspect of this invention, it has been discovered that the quality of pearlescence is affected by the method of production. To this end a further embodiment of the present invention provides a method of preparing a pearlescent concentrate comprising

(i) 60 - 80% wt of a lauryl sulphate salt;

(ii) 12 - 20% wt of a fatty acid amide containing a quaternary ammonium group and a carboxyl radical; and

(iii) 6 - 15% wt of a mixture of ethylene glycol monostearate and ethylene glycol distearate;

in which the precipitation of the crystals is achieved by rapid cooling from the temperature at which solution is achieved to a temperature 15 to 30°C above ambient temperature followed by slow cooling of the liquid to ambient temperatures.

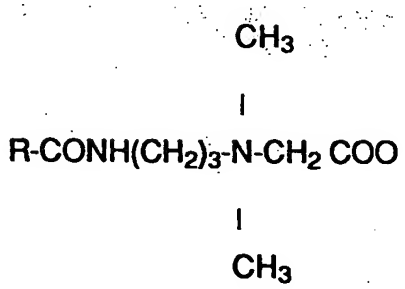
Preferably the ethylene glycol stearate mixture is added to a portion of the lauryl sulphate salt and heated to a maximum of 80°C to form a solution which is then added to a mixture of the fatty acid amide and the remainder of the lauryl sulphate which is at a temperature of not less than 5°C, and most preferably at ambient temperature. In order to achieve rapid cooling in the first stage, the tank to which the hot liquid phase is added, is chilled.

By having an initial rapid quench followed by a slower cooling phase, at a rate of not more than 3°C per minute, the particle size and particle size distribution is controlled to give good pearlescence. In addition, pearlescents of the present invention, prepared according to this method, are translucent in products such as cosmetic, hair, washing and shower preparations, enabling the colour of the finished product to be readily seen. Conventionally, pearlescents give a matt or opaque effect in the finished product so that the colour of the finished product is washed out. In contrast, in pearlescents prepared according to this method, the pearlescence is seen as an adjunct to the colour, enabling the colour of the finished product to be readily seen.

Alternatively, where the strength of colour in the finished product is not important, conventional methods, which result in a flowable product with a pearl-like sheen, without the need for a viscosity modifier, may be used.

According to the present invention, the major component of the pearlescent concentrate is a lauryl sulphate salt, preferably sodium or ammonium lauryl sulphate and most preferably ammonium lauryl sulphate.

Preferably the fatty acid amide is a fatty acid amido betaine. More preferably the fatty acid amide is a fatty acid amido propyl betaine where the fatty acid chain has 12 to 14 carbon atoms. Such mixtures are available commercially as coco amido propyl betaine. Most preferably the fatty acid amide is EMPIGEN BS/AU, which is a coco amido propyl dimethyl betaine of the formula



where R is predominantly C_{12}/C_{14}. EMPIGEN is a registered trade mark of Albright & Wilson Limited.

The present invention is more fully illustrated in the following examples and accompanying drawings. It is to be understood that the scope of the invention may include compositions containing lauryl sulphate salts, fatty acid

amides containing a quaternary ammonium group and a carboxyl radical and ethylene glycol mono- / di- stearate mixtures which have not been specifically illustrated.

In these examples, the viscosity was determined with a Brookfield LV3 viscosimeter at 12 revolutions per minute and at 25°C.

Figure 1 shows the particle size distribution of a pearlescent concentrate according to a preferred embodiment of the present invention, as defined in Example 1.

Figure 2 shows the particle size distribution of a pearlescent concentrate according to a preferred embodiment of the present invention, as defined in Example 2.

Figure 3 shows the particle size distribution of a comparative example of a pearlescent concentrate as defined in Example 3.

Figure 4 shows the particle size distribution of a pearlescent concentrate according to a preferred embodiment of the present invention, as defined in Example 4.

Figure 5 shows the particle size distribution of a pearlescent concentrate according to a preferred embodiment of the present invention, as defined in Example 5.

Figure 6 shows the particle size distribution of a pearlescent concentrate according to a preferred embodiment of the present invention, as defined in Example 6.

Example 1

A pearlescent concentrate according to formula 1 was prepared according to method 1 below. Analysis of the concentrate revealed a narrow particle size distribution as shown in Figure 1 and a viscosity in the range of 2000 to 5000 cps. The concentrate was pearlescent, with a metallic sheen and translucent in the finished product, enabling the colour of the finished product to be readily seen.

Formula 1

	%w/w
EMPICOL 0937	75.00
EMPIGEN BS/AU	15.00
EGMS 3127	8.00
BENZOIC ACID	0.08
SODIUM BENZOATE	0.35
WATER qs to	100.00

EMPICOL 0937 is an ammonium lauryl sulphate based on continuous SO_3 sulphated narrow cut lauryl alcohol marketed by Albright & Wilson Limited. EMPICOL is a registered trade mark of Albright & Wilson Limited. EMPIGEN BS/AU and EGMS 3127 are as hereinbefore defined. Benzoic acid is added as a preservative.

Method 1

PHASE 1:

1. Ensure tank is clean and correctly set-up.
2. Charge water, benzoic acid, sodium benzoate and 55% EMPICOL 0937 (measured in kg) in tank and start stirrer, taking care to avoid aeration.
3. Heat to 75°C and gradually add EGMS 3127 in small portions maintaining temperature at 75°C - 80°C. Mix as required taking care to avoid aeration.
4. Keep stirring until all EGMS 3127 has dissolved. Ensure all EGMS 3127 has dissolved (no lumps).

QUENCHING:

1. Charge remaining EMPICOL 0937 and EMPIGEN BS/AU into main vessel and start stirrer on low speed. Mix until uniform.
2. Apply chilled water to tank to bring temperature of mixture to less than or equal to 20°C.
3. Add Phase 1 from tank, mix with stirrer running moderately fast while recirculating, taking care to avoid aeration.
4. When batch temperature = 45°C, stop recirculation pump and reduce stirring speed to minimum and allow batch temperature to fall to ambient.

NOTE:

1. Citric acid or ammonia to be used of pH adjustment.
2. Do not use salt to reduce viscosity.

5. Make necessary adjustments.

Special Conditions for Process

1. It is essential that Phase 1 be quenched as quickly as possible when ready to perform this step.
2. Liquid phase (ex Phase 1) must be @ 75°C before each cumulative addition of EGMS 3127.
3. Temperature of Phase 1 should not be allowed to exceed 75°C - 80°C, and should be maintained at 75°C - 80°C until ready for transfer.
4. Temperature of cooling water should be as low as practically possible (temperature allowed: ambient).
5. Stirrer is to be left on throughout entire quenching process.

Example 2

A pearlescent concentrate according to formula 1, in which Croda EGMS (experimental), as hereinbefore defined, was substituted for EGMS 3127, was prepared according to method 1. Analysis of the concentrate revealed a narrow particle size distribution as shown in Figure 2. The concentrate had a viscosity of 1,750 cps and a flow pattern pearlescence and translucent in the finished product, enabling the colour of the finished product to be readily seen.

Example 3 (Comparative)

A pearlescent concentrate according to the formula 2, was prepared according to method 2.

Formula 2

		%w/w
EMPICOL 0150		87.41
EMPILAN FD		6.00
HARCROS KEMEST 347		5.00
FORMALIN		0.15
SALT		1.44
EDTA	qs to	100.00

Empicol 0150 is sodium lauryl diethoxysulphate, from Albright & Wilson Limited. Empilan FD is coconut diethanolamide, from Albright & Wilson Limited. Empilan is a registered trade mark of Albright & Wilson Limited. Harcros Kemest 347 is an ethylene glycol monostearate from Harcros Chemical Group having a diester : monoester ratio of 65:35. Salt is added as a viscosity modifier and formalin is added as a preservative.

Method 2

PHASE 1

1. Ensure that the blender is clean and set up correctly.
2. Add to the blender 60% of the Empicol 0150 from bulk tank via batchmeter (measured in kg).
3. Add salt. Turn the recycle pump on to 1 and stirrer to approximately half speed and commence heating.
4. When temperature reaches approximately 50°C add EGMS and continue to heat to 65°-70°C. (Temperature is not to exceed 75°C otherwise final pearl may be poor). Stir for 1 hour and ensure no undissolved EGMS is present.
5. When all the EGMS is dissolved, commence cooling.

QUENCHING

1. When temperature reaches 57° - 58°C turn off pump and stirrer and add remaining 40% of Empicol 0150 from bulk tank via batch meter. Add Empilan FD to 401/B via drum pump while the Empicol 0150 quench material is being added.
2. When all Empilan FD and Empicol 0150 has been added turn on the stirrer to high speed and the pump on 2. (Full cooling should still be on). Temperature should drop rapidly from 50°C to approximately 40°C.
3. When temperature is less than 40°C add formalin and mix for 1 hour.

Analysis of the concentrate revealed a broad particle size distribution as shown in Figure 3. The concentrate had a high viscosity of approximately 90,000 cps resulting in poor flowability and low pumpability. The concentrate had a flat pearlescence and was opaque in the finished product, washing out the colour of the finished product.

Example 4

A pearlescent concentrate according to formula 1, in which Harcros Kemest 347 was substituted for EGMS 3127, was prepared according to method 1. Analysis of the concentrate revealed a broad particle size distribution as shown in Figure 4, a viscosity of 7,000 cps. The concentrate had a flat yellow pearlescence and was opaque in the finished product, washing out the colour of the finished product.

Example 5

A pearlescent concentrate according to the formula of Example 4 was prepared according to method 3.

Method 3

PHASE 1:

1. Charge water, benzoic acid, sodium benzoate and 55% EMPICOL 0937 (measured in kg) in tank.
2. Heat to 70 to 75°C and slowly add Kemest 347.
3. Mix until homogenous and ensure all Kemest 347 has dissolved (no lumps).

QUENCHING:

1. Slowly add mixture of remaining EMPICOL 0937 and EMPIGEN BS/AU.
2. Allow to cool, without assistance, to 40°C.

Analysis of the concentrate revealed a broad particle size distribution as shown in Figure 5 and a viscosity of 8,500 cps. The concentrate was a flat opacifier rather than a pearlescent and was opaque in the finished product, washing out the colour of the finished product.

Example 6

A pearlescent concentrate according to formula 1, in which Henkel Cutina AGS/A was substituted for EGMS 3127, was prepared according to method 1. Henkel Cutina AGS/A is an ethylene glycol distearate from Henkel Australia Pty Ltd having a diester : monoester ratio of 85:15. Analysis of the concentrate revealed a broad particle size distribution as shown in Figure 6 and a relatively high viscosity of 25,750 cps. The concentrate had a uniform white pearlescence.

THE CLAIMS DEFINING THE INVENTIONS ARE AS FOLLOWS:

1. A pearlescent concentrate comprising

(i) 60-80% of a lauryl sulphate salt

(ii) 12-20% of a fatty acid amide containing a quaternary ammonium group and a carboxyl radical; and

(iii) 6-15% of a mixture of ethylene glycol monostearate and ethylene glycol distearate;

prepared under conditions which result in a flowable product with a pearl-like sheen.

2. A pearlescent concentrate according to claim 1, wherein the mixture of ethylene glycol monostearate and ethylene glycol distearate comprises 40% to 60% ethylene glycol distearate and 60% to 40% ethylene glycol stearate by weight.

3. A pearlescent concentrate according to claim 2 containing 44 to 54% by weight ethylene glycol monostearate.

4. A pearlescent concentrate according to any one of claims 2 to 3 having a mean particle size of in the range of 7 to 13 microns and a particle size distribution such that at least 85% of the particles are sized between 5 to 20 microns.

5. A pearlescent concentrate according to claim 4 wherein at least 90% of the particles are sized between 5 to 20 microns.

6. A pearlescent concentrate according to any one of claims 1 to 5 having a viscosity of less than 10000 cps.

7. A pearlescent concentrate according to claim 6 having a viscosity of between 1750 and 6000 cps.

8. A method of preparation of a pearlescent concentrate comprising

(i) 60 - 80% of a lauryl sulphate salt

(ii) 12 - 20% of a fatty acid amide containing a quaternary ammonium group and a carboxyl radical; and

(iii) 6 - 15% of a mixture of ethylene glycol monostearate and ethylene glycol distearate; including

(a) dissolving the mixture of ethylene glycol monostearate and ethylene glycol distearate in a hot solution containing at least 40% of the total amount of lauryl sulphate salt; and

(b) precipitating crystals in the solution by rapidly cooling the solution to a temperature 15 to 30° above ambient temperature followed by slowly cooling the solution to ambient temperatures.

9 A method of preparation of a pearlescent concentrate according to claim 8, wherein the temperature of the hot solution does not exceed 80°C.

10. A method of preparation of a pearlescent concentrate according to claims 8 or 9, wherein the rate of slow cooling does not exceed 3° per minute.

11. A pearlescent concentrate prepared according to the method of any one of claims 8 to 10.

12. A pearlescent concentrate according to claim 11, wherein the mixture of ethylene glycol monostearate and ethylene glycol distearate comprises 40% to 60% ethylene glycol distearate and 60% to 40% ethylene glycol stearate by weight.

13. A pearlescent concentrate according to claim 12, wherein the concentrate contains 44 to 54% by weight ethylene glycol monostearate.

14. A pearlescent concentrate according to any one of claims 11 to 13, wherein the concentrate has a mean particle size of in the range of 7 to 13 microns and a particle size distribution such that at least 85% of the particles are sized between 5 to 20 microns.

15. A pearlescent concentrate according to claim 14 wherein at least 90% of the particles are sized between 5 to 20 microns.

16. A pearlescent concentrate according to any one of claims 11 to 15 having a viscosity of less than 10000 cps.

17. A pearlescent concentrate according to claim 16 having a viscosity of between 1750 and 6000 cps.

DATED this 29th day of March, 1995.

ALBRIGHT & WILSON (AUSTRALIA) LIMITED

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ABSTRACT

A low viscosity pearlescent concentrate comprising

(i) 60 - 80% wt of a lauryl sulphate salt;

(ii) 12 - 20% wt of a fatty acid amide containing a quaternary ammonium group and a carboxyl radical; and

(iii) 6 - 15% wt of a mixture of ethylene glycol monostearate and ethylene glycol distearate, preferably in the ratio of 60 to 40% by weight distearate to 40 to 60 % by weight monostearate;

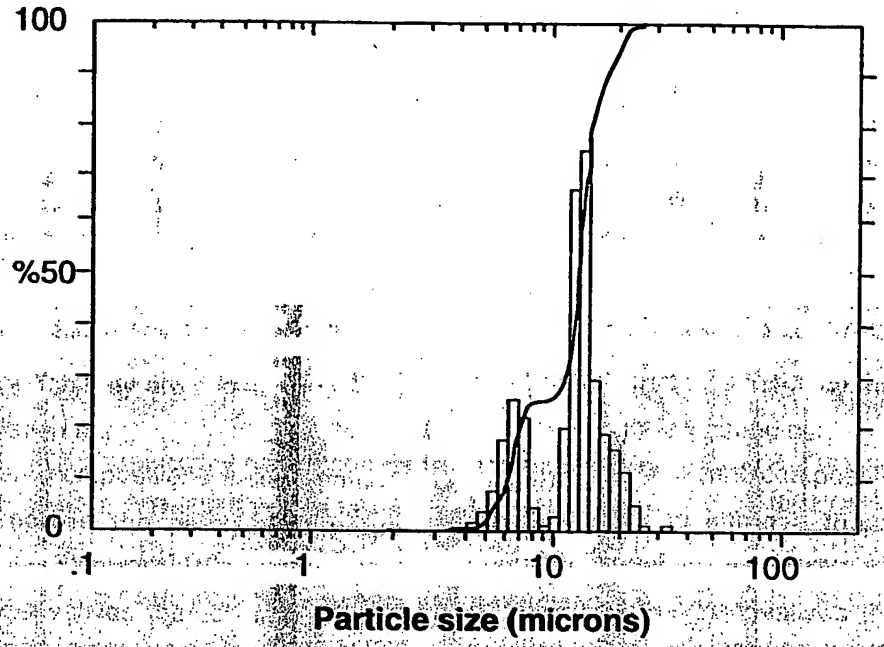
for use in cosmetic preparations, hair and washing or shower preparations. The concentrates have a range of pearlescent sheens, are not contaminated by nitrosamine or 1,4-dioxane and are preferably prepared without the addition of a viscosity modifier. The concentrates are also preferably translucent in the finished product enabling the colour of the finished product to be readily seen.

Preferably the concentrates are prepared by

(a) dissolving the mixture of ethylene glycol monostearate and ethylene glycol distearate in a hot solution containing at least 40% of the total amount of lauryl sulphate salt; and

(b) precipitating crystals in the solution by rapidly cooling the solution to a temperature 15 to 30° above ambient temperature followed by slowly cooling the solution to ambient temperatures.

Fig 1.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Particle size (microns)

Fig 2.

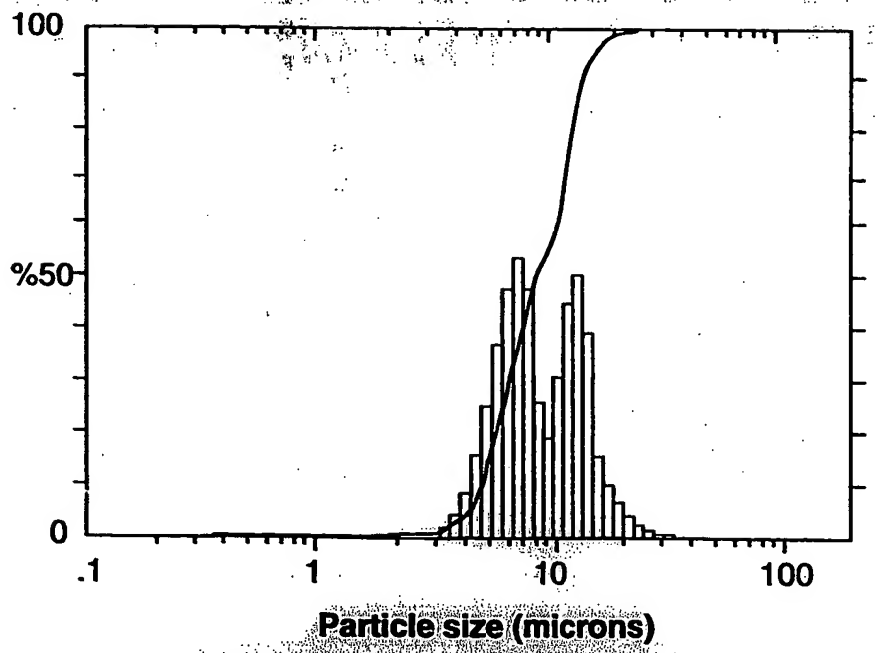
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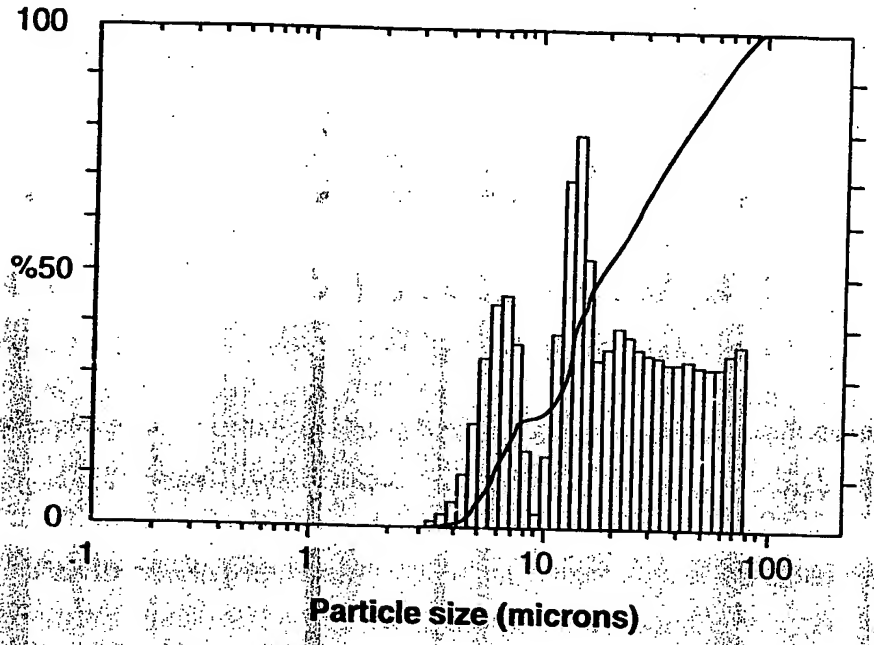
Fig 3.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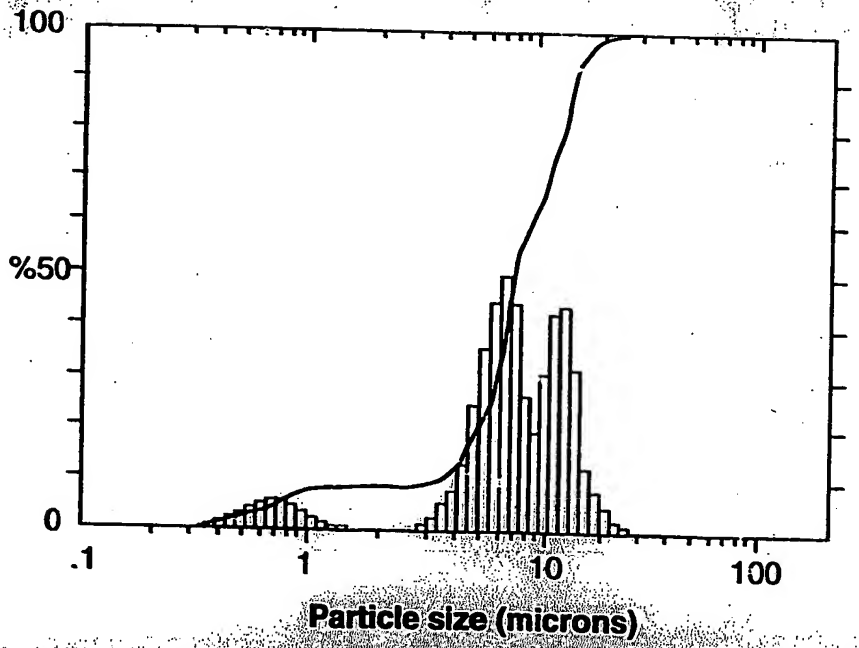
Particle size (microns)

Fig 4.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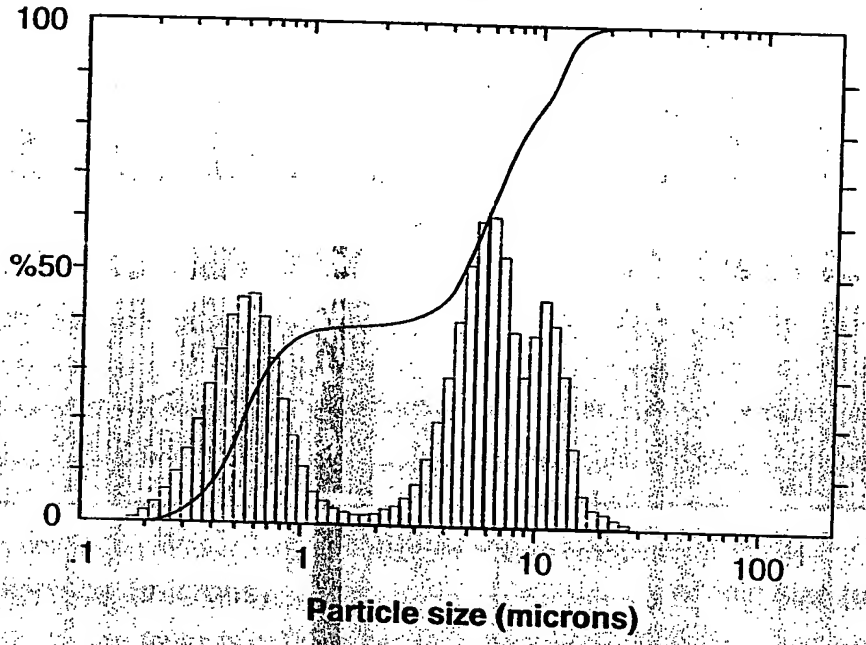
Particle size (microns)

Fig 5.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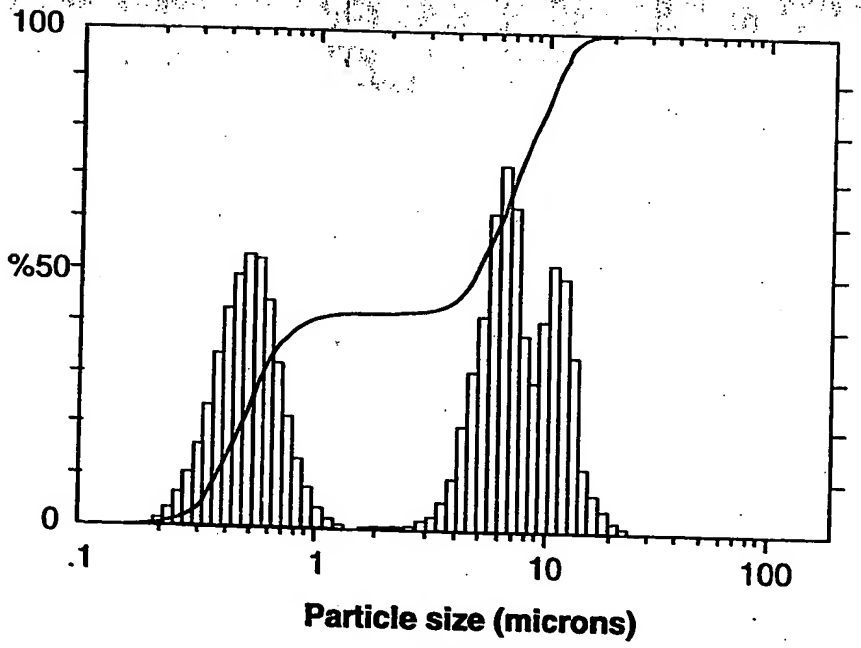
Particle size (microns)

Fig 6.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